CHARACTERIZATION AND PROPERTIES OF SELECTED BIOMASS NANOFILLER REINFORCE ADVANCED CERAMIC

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LIST OF ABBREVIATIONS

CNF	Carbon Nanofiller
BM-CB	Bamboo Carbon Black
BG-CB	Bagasse Carbon Black
OPA-CB	Oil Palm Ash Carbon Black
NBM-CB	Bamboo Nano Carbon Black
NBG-CB	Bagasse Nano Carbon Black
NOPA-CB	Oil Palm Ash Nano Carbon Black
SEM	Scanning Electron Microscopy
SEM-EDX	Scanning Electron Microscopy – Energy Dispersion of X-Ray
TEM	Transmission Electron Microscopy
XRD	X-Ray Diffraction
FT-IR	Fourier Transform Infrared Spectroscopy
TGA	Thermogravimetric Analysis
MC	Moisture Content
CV-IV	Capacitance Voltage-Current Voltage
ESD	Electrostatic Discharge
CTE	Thermal coefficient
MPa	Mega Pascal
Nm	Nano-Meter
Al_2O_3	Alumina
SiO_2	Silica Dioxide
C	Carbon
H	Hydrogen
O	Oxygen
N	Nitrogen
S	Sulfur
GNI	Gross Nation Income

LIST OF PUBLICATION

PENCIRIAN DAN SIFAT-SIFAT KOMPOSIT SERAMIK TERMAJU DIPERKUAT DENGAN PENGISI NANO BIOJISIM KARBON TERPILIH

ABSTRAK

Tujuan kajian ini adalah untuk meneroka karbon hitam daripada biojism pertanian dan sisa pertanian untuk menilai pengisi nano karbon (CNF) sebagai bahan dalam aplikasi seramik. Biojisim daripada buluh , hampas tebu dan abu kelapa sawit digunakan sebagai pendahulu untuk menghasilkan pengisi nano karbon hitam. Relau pirolisis dijalankan pada 800°C dan diikuti dengan pemprosesan bebola -kilang untuk mendapatkan CNF dalam lingkungan 50 nm 100 nm. CNF ditambah kepada alumina dalam pelbagai peratusan berat badan matrik dan tertakluk kepada pensinteran vakum pada 1400°C untuk menghasilkan komposit nanobioseramik. Sifat fizikal, kimia, terma dan morfologi CNF dan komposit nanobioseramik dikaji. Ciri-ciri fizikal kekerasan Vickers, patah kekuatan dan spesifik graviti dianalisakan untuk komposit. Analisis kimia dijalankan dengan analisis pH dan jelmaan Fourier inframerah (FT-IR) untuk CNF. Analisis terma telah dikaji dengan penganalisis Termogravimetri (TGA) untuk CNF dan komposit. Kajian morfologi pada komposit dan CNF dijalankan dengan mikroskop imbasan elektron (SEM), mikroskop elektron transmisi (TEM) dan sinar-X (XRD) untuk CNF dan komposit. Analisis kekonduksian terma, kekonduksian elektrik dan pelepasan pengukuran elektrostatik (ESD) dijalankan ke atas komposit nanobioseramik.

Peratusan karbon telah diterangkan, seperti berikut: BM- CB > BG- CB > OPA -CB oleh analisis EDX. Kesan penambahan CNF ke atas kekerasan Vickers (HV20) menunjukkan trend penurunan untuk setiap siri CNF, disebabkan oleh kesan mikrostruktur menyematkan dalam punca alumina dengan saiz butiran nano. Begitu juga, graviti spesifik (SG) dan ketahanan patah berkurangan apabila berat % CNF

bertambah. Diperhatikan bahawa alumina CNF seramik komposit menjadi lebih ringan dan lebih rapuh dengan muatan CNF bertambah. Penurunan yang ketara diperhatikan apabila muatan meningkat daripada 0.05% kepada 0.5%, yang membuat komposit seramik lebih ringan daripada alumina asal. Keputusan TGA tidak menunjukkan sebarang perubahan penting dalam kestabilan terma antara CNF berbanding dengan karbon hitam. Tiada perubahan ketara komposit nanobioseramik berbanding dengan alumina asal. Keberaliran haba bagi komposit CNF alumina meningkat dengan kandungan CNF. Pengembangan haba (CTE), tidak ketara atau berkelakuan "null" sama seperti alumina asal dengan peningkatan CNF. Komposit nanobioseramik juga tidak menunjukkan sebarang perubahan ketara berbanding dengan alumina asal. pH dan analisis FT-IR, memberi hampas tebu,lebih asid dan buluh bersifat alkali. Dari segi morfologi, komposit nanobioseramik bergantung kepada struktur "grain bounary" CNF and matriks alumina, yang memberikan struktur perpaduan tidak sekata "cohension".

Analisis elektrik pada I-V menunjukkan peningkatan CNF berat. % ke dalam matriks alumina, kenaikan linear diperhatikan. CNF berat 1% dan 0.5 %, CNF seramik komposit mengendalikan hampir 3.0E -10 mA arus berbanding dengan 0.05% dan 0.1 %. Kewujudan CNF di dalam alumina memberi sifat baru komposit nanobioseramik, mengekalkan pengembangan haba tetapi meningkatkan ciri-ciri elektrik. ESD memberikan ramalan dari "insulative" alumina ke "static-dissipative"dengan menambah CNF. Keputusan dapat disimpulkan dengan pemahaman yang jelas terhadap interaksi CNF ke atas mikrostruktur dan sifat bagi komposit nanobioseramik. Ciri-ciri diperhatikan daripada kajian ini akan memberi penambahan ke atas penggunaan teknologi fabrikasi maju dan untuk memaksimumkan manfaat daripada penggunaan sisa pertanian sebagai bahan pengisi nano karbon.

CHARACTERIZATION AND PROPERTIES OF SELECTED BIOMASS CARBON NANOFILLER REINFORCED ADVANCED CERAMIC COMPOSITES

ABSTRACT

The aim of this study was to explore the carbon black nanofillers (CNF) from biomass and agricultural wastes as reinforcement material in ceramic application. Biomass from bamboo, bagasse, and oil palm ash was used as the predecessor for producing carbon black nanofillers. Furnace pyrolysis were carried out at 800°C and followed by ball-mill processing to obtain CNFs in the range of 50 nm to 100 nm. The CNFs were added to alumina in varying weight fractions and subjected to vacuum sintering at 1400°C to produce nano bioceramic composites. The physical, chemical, thermal and morphology properties of CNFs and the nano bioceramic composite were studied. The physical property of Vickers hardness, fracture toughness and specific gravity was analysed for the composite. The chemical analysis conducted with pH analysis and fourier transform infrared (FT-IR) for CNFs. The thermal analysis was studied with thermogravimetric analyser (TGA) for CNFs and composite. The morphology studies on the composite and CNF conducted with scanning electron microscope (SEM), transmission electron microscope (TEM) and X-ray diffraction (XRD) for CNFs and composite. The nano bioceramic composite was further analysed for thermal conductivity, electrical conductivity and electrostatic discharge (ESD) measurement.

The percentage of carbon was described respectively, as followed: BM-CB > BG-CB > OPA-CB by EDX analysis. The effect of CNFs' loading on the Vickers hardness (HV20) shows a decrease trend for each series of CNFs, due to the

microstructure pinning effect in alumina caused by the nano grain size of CNFs. Similarly, the specific gravity (SG) and fracture toughness decreased as the CNFs' wt.% increased. It was observed that alumina-CNF ceramic composite becomes lighter and more brittle as the CNFs' loading increases. The TGA results showed no significant changes in thermal stability between the CNFs compared to carbon blacks. Similarly, the nano bioceramic also showed no significant changes compared to neat alumina. Thermal conductivities of the CNF-alumina composites increased with CNFs loading. The trend in the thermal expansion (CTE), was not significant or behave null same as neat alumina with the increasing of CNFs. The pH and FT-IR analysis on CNFs, revealed bagasse was more acid and bamboo was alkaline in nature. In terms of morphology, the bio nano composite structure was much dependable on the grain structure boundary of CNFs and alumina matrix, which gave an uneven cohesion structure.

The electrical analysis on I-V showed an increase of CNF wt. % into the alumina matrix and gave a linear increment in current. At CNF wt 1% and 0.5% the CNF ceramic composite conducts close to 3.0E-10 mA of current compared to 0.05% and 0.1%. The existence of CNFs into alumina maintains the thermal expansion but improves the electrical characteristics. The ESD properties gave a predictable result from insulative nature of alumina to static-dissipative nature of the nano bioceramic with increase of CNF wt. %. The results obtained are highly encouraging and gave a vivid understanding of CNFs' interaction on the microstructure and properties of the nano bioceramic composite. The observation from this study will give an enhancement on the utilization of advanced fabrication technologies and to maximize the benefit from the agricultural wastes usage as carbon nanofiller materials.

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APPENDIX A

PEER-REVIEWED ARTICLE bioresources.com

Nanobioceramic Composites: A Study of Mechanical, Morphological, and Thermal Properties

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The aim of this study was to explore the incorporation of biomass carbon nanofillers (CNF) into advanced ceramic. Biomass from bamboo, bagasse (remains of sugarcane after pressing), and oil palm ash was used as the predecessor for producing carbon black nanofillers. Furnace pyrolysis was carried out at 1000 °C and was followed by ball-mill processing to obtain carbon nanofillers in the range of 50 nm to 100 nm. CNFs were added to alumina in varying weight fractions and the resulting mixture was subjected to vacuum sintering at 1400 °C to produce nanobioceramic composites. The ceramic composites were characterized for mechanical, thermal, and morphological properties. A high-resolution Charge-coupled device (CCD) camera was used to study the fracture impact and the failure mechanism. An increase in the loading percentage of CNFs in the alumna decreased the specific gravity, vickers hardness (HV), and fracture toughness values of the composite materials. Furthermore, the thermal conductivity and the thermal stability of the ceramic composite increased as compared to the pristine alumina.

Keywords: Ceramic composite, Carbon nanofiller, Thermogravimetric Analysis, Mechanical properties

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INTRODUCTION

Biomass as a precursor of carbon is well known as an active material for energy storage and conversion. Natural lignocellulosic fiber has a high carbon content and good properties. Jute, flax, coconut shells, oil palm, bamboo, bagasse, and oil palm ash are natural fibers that contain high carbon content (Abdul Khalil *et al.* 2009). The preferred source of carbon nanofiller (CNF) in this study is biomass, which is abundantly available as waste from the agricultural activities in Malaysia. Specifically, biomass from bamboo (*Gigantochloa scorechinii*), bagasse, and oil palm ash is generally used as a precursor of carbon black. CNF is extremely porous, has a large surface area, and is typically produced from organic precursors such as bamboo, bagasse, coconut shells, palm-kernel shells, wood chips, sawdust, corncobs, and seeds.

Carbon materials have a naturally high electrical conductivity and low cost, high surface area, porosity, and formability, and possess good chemical and electrochemical resistivity (Mudimela *et al.* 2009). Carbon black is a form of amorphous carbon that has an extremely high surface area to volume ratio. As such, it is one of the first nano-

APPENDIX B

Carbon Nanofiller enhance Ceramic composite: Thermal and Electrical studies.

Sivabalan Sasthiryar,[a] H.P.S. Abdul Khalil,[b]* and Ahmad Z.A.[b]

Study to enhance advance ceramic, alumina (Al_2O_3) with carbon nanofiller(CNF) from oil palm ash (OPA). Oil palm ash used as the predecessor for obtaining carbon black nanofillers through 1000°C furnace pyrolysis with continued ball milling. CNFs in range of 50nm to 100nm is added to alumina by varying weight fractions and sintered at 1400°C to obtain CNF ceramic composite. CNF ceramic composite were subjected to thermal and electrical studies. The thermal expansion based on the incremental of weight of CNF did not give positive advances. The composite behaved null or same as the matrix alumina. The electrical studies on IV showed the weight increase of CNF gave positive results. ESD properties also gave a predictable result from insulative nature of alumina to dissipative with adding CNFs. With the existence of CNFs into ceramic, the nature of new composite maintains the thermal expansion but improves the electrical characteristics.

Keywords: Oil palm ash (OPA), Carbon nanofiller (CNF), Thermal coefficient (CTE), Electrostatic Discharge (ESD), Voltage –current (IV).

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INTRODUCTION

The potential of Oil palm ash (OPA) as a new filler in composites had been studied by several researcher; (Ismail and Haw, 2008), (Ismail and Shaari, 2010), and (Bhat and Abdul Khalil, 2011). The mechanical, thermal, physical and morphological properties of the composites varies upon the incorporation of OPA into the composites system, such as filler loading (Ismail and Haw, 2008); (Ismail and Shaari, 2010), effect of compatiblizer with OPA (Bhat and Abdul Khalil, 2011), and structural size of OPA (Bhat and Abdul Khalil, 2011). OPA is natural fiber which contains high carbon content and this has been inspired with the growing interest in the oil palm waste utilization as a renewable source of energy. In this study OPA as precursor of carbon black is a form of amorphous carbon has an extremely high surface area to volume ratio(Prasantha et al., 2009), and as such it is CNFs. As known, carbon materials have high electrical conductivity, low cost, high surface area, porosity, formability and possess good chemical and electrochemical resistivity (Prasanth et al., 2009). This also support by

CHAPTER 1
INTRODUCTION

1.1 Introduction

Nano biocomposites are nanometer scaled sized biofillers from biomass that gives unique advantages over traditional inorganic nanoparticles. The nature of nano sized biofillers exhibit the predominant advantages such as easily available, low density, have high specific strength, high rigidity and superconductive properties (Andreoni, 2010). This gives considerable interest in application and research studies of its characteristics. Significant improvement in terms of mechanical, barrier and thermal properties have been reported in such nano biocomposites (Alan *et al.*, 2009). Moreover, nano-composites have been used in application of high voltage electrical insulation system and many studies reported on the influence of nano-composites in terms of dielectric properties (Alexandre and Dubios, 2000).

Carbon as an active material for energy storage and conversion is well known to be the precursors of the natural biomass. Biomass based nanofillers consist of the rod-like whiskers of cellulose and chitin, the platelet-like nanocrystals of starch, the self-organized nanophase of supramolecular lignin complexes and many artificial nanofillers derived from biomass (Chang *et al.*, 2011).

The carbon nanotechnology discovery begins with fullerenes carbons in 1985 and continues with carbon nanotube in the year 1991(Andreoni, 2010). The main inhibitor of the carbon nanotechnology growth was the production cost. The advancement in combustion methods for producing fullerenes reduced the production cost by at least one tenth. This enhancement was accelerated with the novel discovery of carbon nanotubes (CNTs) and nanofillers (CNFs) material (Andreoni, 2010). This

material has unique geometrical structure, stable mechanical and chemical properties. Research study by Reynolds et al., (2010), estimated, the market for carbon nanotube was worth $100 million in the year 2007 and will be increasing of it production with next 5 years as many more commercial carbon nanomaterial produce for industrial applications.

Since the growth of the high performance nano-composite materials for engineering applications under high demanding working conditions has been increasing, the safety endurance and economic efficiency of these composites must have excellent mechanical and tribological properties. They also need to have low specific weight and high resistance to degradation. Due to their good mechanical performance and low density, these composites are considered highly for electrical conductivity applications (Alan *et al.*, 2009).

Thus, industrial applications often introduce new usage of composite materials which in return, covers some of the demanded properties such as mechanically reinforcing fillers or fibers (glass, carbon and aramid) or particles such as ceramic powders (Arivalagan, 2011). The nanoparticles' matrix deformation less than micro particles and integrate better into the composite microstructure as they approach to molecular dimensions. The strong interactions with the matrix, can be expected that the nanoparticle influences the deformation mechanisms in the composite matrix on the microscale and the nanoscale (Arivalagan, 2011). Nanoparticles have good dispersion in the matrix which results in a more uniform stress distribution and also minimizes the presence of agglomerates which acts as stress concentration centers. This will increase the general strength and modulus of the composite totally.

In this study, sources of carbon nanofiller (CNF) from biomass which is abundantly available waste from the agricultural activities in Malaysia is used as

nanoparticles. Naturally lignocellulosic fiber contains high carbon content with good properties (Abdul Khalil *et al.*, 2009). The potential of Oil palm ash (OPA) as a new filler in composites had been studied by several researcher (Ismail and Haw, 2008), (Ismail and Shaari, 2010) and (Bhat and Abdul Khalil, 2011). The OPA act as a precursor of carbon black is a form of amorphous carbon has an extremely high surface area to volume ratio (Prasantha *et al.*, 2009).

In Malaysia, the biomass abundant including empty fruit branches (EFB), coconut shells, oil palm waste and others were produced about few million tons metric a year (Abdul Khalil and Rozman, 2004). Specifically, the biomass from bamboo (*Gigantochloa scoretechinii*), bagasse and oil palm ash is used as a precursor of carbon black. The CNF is extremely porous with large surface area, and typically produced from organic precursors such as bamboo, bagasse, coconut shells, palm-kernel shells, wood chips, sawdust, corncob and seeds (Noriman, 2005).

Currently, in advance ceramic the nanoparticles used to strengthen are mainly inorganic. With the successive introduction of CNFs from biomass to strengthen advance ceramic such as alumina, zirconia, silicon nitride, alumina nitride, boron nitride and etc. enhances the electrical properties of insulating ceramics. This will allow electrical discharge and other properties of the ceramic to be either preserved or even improved. This advanced nano composite materials have gained popularity for wide engineering applications. Along with their unique mechanical, thermal, and electrical properties, this improves all types of products and commercialization of products that exploit within. Many researches have also demonstrated the use of nano structural materials as reinforcements, such as nano apatite, nanoclay and nanofibers(polymer-based or carbon nanotubes) to enhance the mechanical properties and thermal stability (Alan *et al.*, 2009). The use of a carbon source has improved the thermal shock

resistance of the refractory due to the low thermal expansion, high thermal conductivity, and low modulus of elasticity in composites. On the other hand, these composites have a few disadvantages, such as oxidation (Amin *et al.*, 2009). The CNT/CNF reinforced cement composites are able to provide electromagnetic interference shielding (Fu and Chang 1996) and also capable of non-destructive flaw detection (Chen and Chung, 1993). Utilization of CNTs/CNFs has been investigated for applications in electronic and construction industry although the expected improvement has not been fully achieved (Li *et al.,* 2005). Moreover, the electrical conductivity can be imparted by having the dispersed CNTs/CNFs particles direct into contact by making a continuous phase of conductive pathway (Al-Saleh and Sundararaj, 2008).

The addition of CNF greatly enhances the electrical properties of insulating ceramics allowing electrical discharge machining to be used to manufacture intricate parts. This new opportunity of advance ceramic parts with adding CNF can be manufactured for the engineering and biomedical applications. Since the CNFs discovery, a great deal of attention has been given due to their exceptional properties (Calvert, 2009; Zhu *et al*, 2004; Makar and Beaudoin, 2004). This attention also includes their usage as nanofillers in ceramic materials in order to develop tougher composites (Weing *et al.*, 1997).

This study has been carried out, by varying loading percentage of CNF in alumina matrix (ceramic). The morphological study was further conducted to understand the interaction between the CNF and alumina matrix. Once the series of composite mixture is obtained, the mechanical, electromechanical, and nano-structural studies are carried on to understand the characteristics of nanofillers in advance ceramic. This aimed to enhance current and future applications in advance industries such as electronic, microelectronic, aerospace and etc.

1.2 Problem Statement

The aim is an exploration to incorporate biomass nanofillers carbons into advance ceramic matrix. This study is to understand the bio-agricultural waste as a subsequent replacement fillers as in nanofillers for advance ceramics which currently inorganic fillers has been use to gain engineering application. The study also aimed on the bio waste carbon particularly due to low cost comparability among the industrial inorganic fillers. The abundant availability and feasible to gain engineering application mainly in thermal and electrical conductivity is an important factor of carbon nanofillers in this study. Naturally this study requires scientific approach in terms of qualitative and qualitative evident. The lack of detail sufficient scientific information about the utilization of biomass carbon nanofillers in advance ceramic is the novelty exploration in terms of characteristics and future application.

1.3 Scope of Work

The scope of research is to investigate the usage of natural bio mass and agricultural waste added to advance ceramic to give an industrial application and a novel in nano materials studies to reinforce current and future commercial products. This combination of unique properties of CNFs and advance ceramics is brought together to produce biomass nanofiller reinforce advance ceramic. Physical, mechanical, thermal and electrical analysis conducted to discover the unseen possibilities of creating new engineered materials for productive applications. As known, today CNF composites products are used as electrically conductive additives in automotive plastics for automotive fuel systems requiring antistatic properties. The CNF composites are known for exhibiting unique mechanical, electrical and thermal

properties. They are very useful for a wide range of engineering applications in materials.

In semiconductor industry, the CNF-based products procure very promising additives. Thus, they have a clear competitive advantage since the trends in electronics towards smaller and smaller components will only be possible with cleaner materials. Their fibrous structure which finely dispersed in materials gives a good electrically conductivity. This increases the number of electrical contacts and ensure a smoother flow of electrons. Therefore, a lower content of additive is needed to give good resistivity, compare to inorganic fillers. The comparison between carbon fillers and metals, during designing antistatic materials meant to use in clean room environment, demands the decreasing probability of contaminating microchips and other sensitive components. Thus, this study is an exploration to further understand the benefits of CNF reinforce advance ceramic in terms of mechanical properties, electro thermal and electrical conductivity.

1.4 Objectives

This research study is planned to carry out the following objectives:

1. To characterizes the properties of selected biomass carbon black nanofiller of bamboo, bagasse and oil palm ash;
2. To investigate the differences of varying loading percentage of carbon black nanofiller from the selected biomass on alumina matrix composite and to study the mechanical and electro thermal properties;
3. To study the morphological interaction between the carbon black nanofiller and alumina matrix.

1.5 Organization of Thesis

This thesis has been divided into five (5) chapters. The brief description and contents of this thesis (chapter-wise) are given below.

Chapter 1: This chapter gives the introduction of the research work. It illustrates the background, problem statement, scope of work of the research and describes the objectives for this research study.

Chapter 2: In this chapter, literature review of the research study is given. Review of biomass material, composite, carbon black, carbon nanofillers and application of CNF-ceramic composite.

Chapter 3: This chapter consists of the materials and methods used in this research study. Thus, illustration of raw material used in the preparation of carbon black, predecessor for CNFs and the analysis of carbon black along with CNFs characteristics. The final product, nano-biocomposite (CNF+Alumina)'s preparation through sintering process. The analysis conducted on the nano-biocomposite includes physical, mechanical, thermal and electrical properties.

Chapter 4: This chapter reports, all the results and discussion conducted from the analysis. Carbon black, CNFs and nano biocomposite analysis results and discussion in agreement with the fulfillment of the objectives of this research study.

Chapter 5: In this chapter, conclusion and recommendation is given. The overall conclusion with the nano-biocomposite is summarized and future recommendation is also suggested.

CHAPTER 2
LITERATURE REVIEW

2.1 Biomass

Earth's vegetation which covers the planet is a natural storage of solar energy. The organic matter composing it is called biomass. Biomass is produced through the photosynthesis process, with the combination of carbon dioxide from the atmosphere and underground water. The final product of photosynthesis are sugars, starch, cellulose, lignin, protein substances, fats, etc. The same solar energy that activated the photosynthesis is contained in the chemical bonds of these substances. This translates to 2×10^{11} tons of carbons are fixed each year, with a corresponding energy content of 70×10^{3} megatons of oil (Justin and Ianthe, 2012).

Once the biomass undergoes burning, oxygen form the atmosphere combines with the carbon contained in the biomass, thus generating energy in the form of heat. It also freeing carbon dioxide and water to the nature. Carbon dioxide goes back into the atmosphere and will be re-used in the photosynthesis process to produce new biomass (Melssen, 2013). Hence these recycle process makes biomass is a renewable resource. Generally, biomass indicates several types of products: agricultural, forest residues, waste of wood processing industry, food residues, urban waste, production of fuel waste and other types of heterogeneous industrial waste (Melssen, 2013). In additional the scope has been extended to extending include all types of biomass from sources such as rubber, wood and rice husk (Ludovic and Sanjeeb, 2004).

The world biomass production estimates in 104.9 petagrams (104.9×10^{15} g) of carbon per year. This is concentrated half in the ocean and half on land (Alan and

Andrew, 1997). Traditionally, humans have harvested biomass energy ever since fire was introduced to humans (Justin and Ianthe, 2012). At present era, biomass is popular source of fuel for domestic use in many developing countries.

In Malaysia, currently about 12 percent of Gross Nation Income (GNI) is generated from the agriculture sector. This value is a significant amount of biomass generated every year across a variety of crops. The palm oil is the largest contributor to GNI within agriculture sector, contributing about 8 percent or over RM 80 billion. Nevertheless the palm oil activities, generates the largest amount of biomass. It is estimated in 2012, at 83 million tons. It is expected to increase to about 100 million dry tons by 2020 (Melssen, 2013). The National Biomass Strategy 2020, had thus far focused on oil palm biomass. The oil palm biomass generated today is generally returned to field to release its nutrients and replenish the soil, as organic fertilizer. This biomass, organic fertilizer plays an important role to ensure the sustainability and potential high value usage as bioenergy, bioethanol and bio based chemicals. (Ludovic and Sanjeeb, 2004).

By 2020, an additional of 20 million tonnes of oil palm biomass is expected to be utilized for higher-value uses and could have a significantly contributed to the nation's economy. Thus, this translates to a significant incremental addition of RM 30 billion by 2020 (Melssen, 2013). This National Biomass Strategy 2020 offers Malaysia a way to meet its renewable energy target, reduce emissions and create about 66,000 incremental jobs. It also offers an opportunity strategy for Malaysia to build several biofuels (pellets and ethanol) and bio based chemical downstream clusters and ensure the nation benefits from the potential value creation (Melssen, 2013).

On contrary, from a supply chain perspective, by 2020, Malaysia's palm oil industry is expected to generate about 100 million dry tons of solid biomass which

includes the empty fruit bunches (EFB), mesocarp fibres (MF), palm kernel shells (PKS), the oil palm fronds and trunks (Rahman *et al.,* 2006). A huge amount of this solid biomass will be in the plantations as fertilizer. A small fraction amount will be utilized for bioenergy and as an introduction to renewable energy (Melssen, 2013).

Mostly unused palm oil mill effluent (POME) will be converted into biogas for either powering the mills or selling power. This activities will add into the national grid and would contribute towards the renewable energy target of Malaysia, 410 MW installed biogas capacity by 2030 (Melssen, 2013).

This initiative alone would reduce the nation's carbon dioxide (CO_2) emissions by 12 percent. Therefore, it will also free up significant biomass for higher value usage. This combination gives the potential of freeing up to 20–30%, the available solid biomass for higher value added uses without affecting oil palm yields (Ioannidou and Zabaniotou, 2007). The successful realization of National Biomass Strategy is strongly depending upon collaboration among many government agencies with biomass production owners in Malaysia. This will require the support from the private sector and academically research institutions (Melssen, 2013).

2.1.1 Bamboo

Bamboo fibre is also a good candidate of non-wood fibres and can be exploited for the design and development of polymer composites. It is found in abundance in Asia and South America. In many Asian countries, bamboo (Figure 2.1) has not been explored fully to its extent although it is considered as natural engineering material. This sustainable material has evolved as backbone for socio-economical status of society as it takes several months to grow up. Traditionally bamboo has been used in various living facility and tools, which owes to its high strength to its weight. Due to the extra lignin

content covering the bamboo fibres, it is non-brittle in nature as compared to other natural fibre and possessed finer mechanical properties (Abdul Khalil et al. 2012c).

Six countries in Asia which are India, China, Indonesia, Philippines, Myanmar, and Vietnam has the largest area of bamboo vegetation. Almost 11.4 million hectares are from India and 5.4 million hectares are from China and 2 million hectares are from Indonesia. India and China are reported as the largest area which approximately 70 percentage of total area of bamboo in Asia. The bamboo area in Asia has increased by 10 percentages over the last 15 years, primarily due to large-scale planting of bamboo in China and to a lesser extent in India (Lobovikov et al. 2007).

Bamboos range in size from low, shrubby forms only ten feet (3 m) tall to towering giants over 100 feet (30 m). Aerial stems (called culms) develop from scaly, underground stems called rhizomes that bear roots at the nodes where the leaf like scales are attached (Clark, 2003).

Figure 2.1: Bamboo

The supply of bamboo from natural stands is abundant in Peninsular Malaysia. The extent of natural bamboo stands in Peninsular Malaysia was estimated about 400,000 hectares. They are mostly found in ex-logging areas in the states of Kelantan, Terengganu, Pahang, Perak and Negeri Sembilan. The dominant species is *Gigantochloa scortechinii* that has large culm diameter (Abdul Razak, 1989)

The selection of bamboo species for various applications is not only related to physical and mechanical properties but also to the chemical composition (Tomalang et al., 1980). The chemical composition of bamboo varies according to species, conditions of growth, age of bamboo and the part of the culm. Cellulose, hemicellulose and lignin make up the main constituents of bamboo with resins, tannins, waxes and inorganic salts as minor constituents. Bamboo normally consists of about 50-70% holocellulose with 30% pentosans and 20-25% lignin (Tamalong *et al*., 1980; Chen *et al.*, 1985) According to Amanda *et al.* (1996), bamboo contain 44.5% cellulose, 20.5% lignin, 32% soluble matter, 0.3% nitrogen and 2% ash. Li *et al*. (2007), in their study on *Phyllostachyas pubbescens* bamboo species found that the average relative proportion of the main chemical constituents are holocellulose (71.4%), α-cellulose (47%), klason lignin (22.8%), alcohol-toluene soluble extractive (5%) and ash (1.5%).

The proportion of lignin and carbohydrates in bamboo changes within the first year with the culm maturation when the soft and fragile sprout becomes hard and strong. However, after full maturation, the chemical composition tends to remain constant. The nodes contain less water-soluble extractives, ash and lignin but more cellulose than the internodes. The season influences the amount of water-soluble materials present, which are higher in the dry season than in the rainy season. The starch content reaches its maximum in the driest months before the rainy season and sprouting (Liese, 1985). The ash content (1-5%) is higher in the inner part than in the outer one. The silica varies

on an average from 0.5 to 4%, increasing from bottom to top. Most silica is deposited in the epidermis, whereas the nodes contain little silica and the tissues of the internodes almost none. Silica content affects the pulping properties of bamboo (Abdul Khalil et al. 2012c). It is important to understand the physical and mechanical properties of bamboo in order to effectively utilize bamboo as a material industrial or structural applications in Malaysia (Abdul. Latif, 1991).

Table 2.1 illustrated the chemical composition in bamboo, studied by Higuchi, 1955. The study on the physical characteristics of bamboo indicated that bamboo generally tapers from the base towards the tip with significant decrease in culm diameter, girth, internode length and culm wall thickness. It reaches its maximum height within a year.

Table 2.1: Chemical analysis of bamboo (Higuchi, 1955)

Component	Value, %
Cellulose	49.1%
Lignin	26.1%
Extractives soluble in alcohol – acetone	4.6%
Pentosan	27.7%
Ash content	1.3%

The bamboo diameter and girth which further implies that bamboo acquires its full diameter during sprouting and thus attains its final diameter without showing any secondary thickening (Ueda, 1981; Liese, 1985b; Chaturvedi, 1988). The culm literally consists of nodes and internodes with cells in the internodes axially oriented while at the nodes they are transversely orientated. The epidermal layer of the internodes are generally made up of a highly lignified and cutinized layer with a wax coating on top but the inner layer is made up of numerous sclerenchyma cells. In transverse section, the anatomical structure is determined by the vascular bundles present with the one at the

peripheral normally appearing small but numerous. On the other hand, vascular bundles found in the inner part are larger but fewer (Liese, 1985a).

Brown *et al.* (1952) reported that the wood of many tree-species (particularly in coniferous tree), decreases in basic density with increasing distance above the ground. However, this is the opposite of bamboo where the density increases with height while the moisture content decreases. The variation in physical properties is associated with the anatomical structure of bamboo (Liese and Grover, 1961; Sharma *et al.,* 1972). Many inconsistent results were however reported with regards to density relationships due to its variable properties. The physical properties such as moisture content, specific gravity and volumetric shrinkage in bamboo; *Dendrocalamus hamiltonii* are significantly affected by the height position and presence of nodes. The internodes of the culm were found to have higher moisture contents and volumetric shrinkage with lower specific gravity (Kabir *et al*., 1995).

The node of a bamboo culm consists of the nodal ridge, the sheath scar and the diaphragm. From anatomical studies of bamboo it has been concluded that nodes play an important role in determining the physical and mechanical properties of bamboo; the fiber length increases from the nodes to the center of the internodes. However, it does not occur at constant intervals that causes a problem in design considerations and practice, as joints and/or support are preferably located near nodes for round form applications(Abdul. Latif, 1991). Surjokusumo and Nugroho (1995) reported that the presence of nodes is a disadvantage as a reinforcement material except for the properties of shear and shrinkage.

Ota (1950) was the first to investigate on the influence of the percentage of structural elements on the specific gravity and compressive strength of bamboo splints from the outer and inner layer. Across the culm wall the fiber length often increases

from the periphery towards the middle and decreases towards the inner part. Murphy and Alvin (1992) investigated bamboo; *Physllostachys virideglaucescens* and found that the extent of polylamellation was influenced by the position of the vascular bundle in the culm wall and significantly with position within the vascular bundles. This characteristic pattern of fiber wall lamellation probably influences the mechanical properties and failure characteristics of the material and warrants further investigation.

2.1.2 Bagasse

Bagasse as shown in Figure 2.2 (sometimes spelled *bagass*) is the biomass remaining after sugarcane stalks have been crushed to extract their juice. Sugarcane or Sugar cane (*Saccharum*) is a genus of 6 to 37 species (depending on taxonomic interpretation) of tall grasses (family Poaceae, tribe Andropogoneae). They are native to warm temperate to tropical regions and their fibrous stalks that are rich in sugar and measure 2 to 6 meters tall. Typical commercial varieties grown under normal field conditions have a height of 1.5 to 3 meters and are 1.8 to 5 cm in diameter. The stalk surface can be greenish, yellowish or reddish in color and is covered with a thin waxy layer (Dillewijn, 1952). 30% of bagasse is produced by a sugar factory. These crushed stalks; bagasse consists of 50% cellulose, 25% hemicelluloses, and 25% lignin (Hemmasi *et al.,* 2011). Bagasse represents fibers that are obtained from the stem or stalk of the plants, and not classified as bast fibers (Romanoschi *et al.,* 1998). Chemical composition of sugarcane bagasse is shown in Table 2.2. The arrangement pattern of bast fibers are in a definite ring pattern, while in sugar cane they are more randomly dispersed. Cellulose is a natural linear polymer and has polymer chains of 2000 to 3000 units (Paturau, 1989) with a specific gravity about 1.55 (Elsunni and Collier, 1996).

Cellulose is highly crystalline regardless of the source and the ordered chains are tightly packed, have a strong intermolecular hydrogen bonding because of the preponderance of hydroxyl groups (Romanoschi *et al.*, 1998).

Table 2.2: Chemical composition of sugarcane bagasse. (Hemmasi et al., 2011*)*

Component	Value, %
Cellulose	55.8 %
Lignin	20.5 %
Extractives soluble in alcohol – acetone	3.3 %
Ash content	1.9 %

Huge amount of efforts and research have attempted to utilize bagasse as a renewable feedstock for power generation and for the production of bio-based materials. Bagasse is often used as a primary fuel source for sugar mills by burning in large quantity to produce sufficient heat energy for the sugar mill. A secondary use of bagasse is introduced to cogeneration the use as a fuel source to provide heat energy and electricity (Tewari *et al.,* 2012).

Figure 2.2: Bagasse

The CO_2 emissions from this activities, are equal to the amount of CO_2 that the sugarcane plant used up from the atmosphere during its growing phase, this makes the process of cogeneration appear to be greenhouse gas-neutral (Verma *et al.*, 2012).

Bagasse newsprint paper is a commercially successful in India, Mexico, and Indonesia (Romanoschi, 1998). Bagasse is rich in cellulose content and currently tested for production of commercial quantities such as a "Tree-Free" alternative for making paper. Furthermore, there is no bleaching required in this process and it is more biodegradable, easier to recycle, and overall has less impact on the environment (Chiparus, 2004).

Currently 85% of bagasse production is burnt and the excess is deposited on empty fields to alter the landscape in Brazil. Approximately 9% of bagasse is used in alcohol (ethanol) production and it is a good replacement for the fossil fuels in terms of environmentally friendly fuel. In additional, ethanol is a very versatile chemical in which a variety of chemicals can be produced. In Brazil, ethanol produced from the sugarcane is a popular fuel (Verma *et al.*, 2012).

The Louisiana State University (LSU) conducted a research to determine the feasibility of sugar cane rind fibers for textile and geotextile applications (Elsunni and Collier 1996). A suitable nonwoven mat for geotextiles should sustain, or at least, prevent erosion and at the same time it should be penetrable by growing plants. It also must be capable of permitting interaction between air, soil and allow rain to penetrate (Collier *et al.*, 1995). Thus, the local sugar cane mills can produce a low-cost, biodegradable geotextile which will be providing an economic benefits.

2.1.3 Oil Palm Ash

As the second largest producer and the largest exporter of crude palm oil in the world, Malaysia has accumulated huge amount of biomass (80 million tonnes in 2010) (Choong, 2012). The utilization of oil palm ash (Figure 2.3) as a renewable source of energy or feedstock has been a growing inspiring interest. This is further strengthened and driven by the insight that oil constitutes only 10% of the palm production, while the rest 90% is the biomass. (Foo and Hameed, 2009a).

Figure 2.3: Oil Palm Ash

Currently with the arrival concept of generating energy from oil palm waste, in the forms of palm leaves, OPF, OPT, EFB, PKF, palm fibers and palm stones (Chavalparit *et al.,* 2006), has received stern encourages and considerations worldwide. During the combustion at 800–1000°C from the fired-boiler furnaces, the oil palm ash waste becomes one of the largest readily available and under-utilized biomass resource. The oil palm ash collected from the particulate collection equipment attached upstream to the stacks of oil palm waste fired boilers (Chindaprasirt *et al.,* 2008a).

In Malaysia alone, the potential oil palm ash production is designated at 4 million tons/year (Mohamed *et al.,* 2005). This gives a huge criticisms and complaints, mainly attributed to its persistent, carcinogenic and bio-accumulative effects (Subramaniam *et al.,* 2008). Addition to that, the price of the ash disposal cost (either in landfills or ash ponds) hitting as high as $5/tons in developing countries and $50/tons in developed countries, thus, needs an urgency of transforming the residue into a more valuable end product has been promulgated. Table 2.3 exhibits the composition analysis of the OPA. The raw OPA was evidenced consisting a rather spherical particles with a median size of 183.0 μm while medium and small particles ground palm ash were individually noted containing crushed shape structures with a median of 15.9 and 7.4 μm (Jaturapitakkul *et al.,* 2007).

Table 2.3: The inorganic composition analysis of the OPA

	Chemical constituents Composition(%)						
	(1)	(2)	(3)	(4)	(5)	(6)	(7)
Silicon dioxide	63.6	57.7	65.3	57.8	65.3	57.7	43.6
Aluminum oxide	1.6	4.5	2.5	4.6	2.6	4.6	11.4
Iron oxide	1.4	3.3	1.9	3.3	2	3.3	8.4
Calcium oxide	7.6	6.5	6.4	6.6	6.4	6.6	4.8
Magnesium oxide	3.9	4.2	3	4.2	3.1	4.2	0.4
Sodium oxide	0.1	0.5	0.3	0.5	0.3	0.5	4.7
Potassium oxide	6.9	8.2	5.7	8.3	5.7	8.3	3.5
Sulfur trioxide	0.2	0.2	0.4	0.3	0.5	0.3	2.8
Loss of ignition	9.6	10.5	10	10.1	10.1	10.5	18
SiO_2+Al_2O_3 +Fe_2O_3	66.6	65.5	69.9	65.7	69.9	65.6	63.4

*(Chindaprasirt *et al.*, 2008b)[1], (Tangchirapat *et al.*, 2009)[2], (Jaturapitakkul *et al.*, 2007)[3], (Chindaprasirt *et al.*, 2007)[4], (Chindaprasirt *et al.*, 2007)[5], (Tangchirapat *et al.*, 2007)[6], (Awal and Hussin, 1997)[7]

In most cases, the chemical elements of oil palm ash are found to be silicon dioxide, aluminum oxide, iron oxide, calcium oxide, magnesium oxide, sodium oxide, potassium oxide and sulfur trioxide (Foo and Hameed, 2009a). The varieties of proportion of irrigated area, geographical conditions, fertilizers used, climatic variation, soil chemistry, timeliness of production and agronomic practices in the oil palm growth process contributes to the chemical elements' fluctuation in OPA.

Currently the throwaway waste from the fired-boiler furnaces, Oil palm ash has emerged as an ideal adsorbent in the wastewater treatment processes and as air purifier (Dahlan *et al.,* 2007). Several researchers had working with OPA as novel adsorbents for different applications; (Mohamed *et al.,* 2005; Zainudin *et al.,* 2005; Chu and Hashim, 2002; Ahmad *et al.,* 2007; Hameed *et al.,* 2007; Hasan *et al.,* 2008). The finding provides a two-fold advantage with respect to environmental management. First, by converting to useful and value-added adsorbents, the huge loads of oil palm waste can be reduced. Second, with the development of low-cost adsorbent, it may overcome the wastewaters and air pollution at a reasonable cost. This will solve a part of the global agricultural refuse and wastewater treatment problem (Foo and Hameed, 2009b).

2.2 Carbon Black (CB)

Carbon is a very important component of all known living systems, and without it life as we know it could not exist (Bird and Gro¨cke, 1997). It is also found in abundance in the sun, stars, comets, and atmospheres of most planets (Kuhlbusch and Crutzen, 1996). Carbon was discovered in prehistory by ancient humans, who manufactured it by burning organic material in insufficient oxygen (making charcoal). In the current age, the major economic use of carbon is in the form of hydrocarbons, most notably the fossil fuel methane gas and crude oil (petroleum). Crude oil forms the raw material for many synthetic substances, many of which are collectively called plastics (Liang *et al*., 2008).

The name carbon black is generally used as a generic name for those blacks that are made from the partial burning or carbonizing of natural gas, oil, wood, vegetables and other organic matter. On the basis of a number of factors such as low production cost and purity these carbon blacks can be a starting material for carbon nanotechnology (Koo, 2006). Since carbon absorbs light so well, it often appears dark with infrared imaging, revealing an artist's charcoal sketch under the painting (Haberstroh, 2006).

Carbon black (Figure 2.4) is known to have high porosity. Carbon black flame produces infrared rays not visible with our eyes. This warmth promotes proper blood circulation and renewal. Carbon black generates negative ions and used in the parasympathetic nervous system, which relaxes the mind and body system (Kuhlbusch and Crutzen, 1996).

Figure 2.4: Carbon Black (Kuhlbusch and Crutzen, 1996)

Carbon black is a form of para-crystalline carbon that has a high surface-area-to-volume ratio. However, carbon black is widely used as a model compound for diesel soot for diesel oxidation experiments (Kuhlbusch and Crutzen, 1996).

2.2.1 Carbon Black classification

Carbon black is also used as a pigment in inks, paints and coatings and in plastics. Exposures to carbon black vary markedly between and within production facilities and over time. Carbon black classification is based on the application and usage, hence approximate about 95% it's usage in rubber technology (Pries *et al*., 2010). In addition to the huge demand of carbon black introduce into ultra-high technology and industry, hence American Standard of Testing and Measurement (ASTM) have introduce a system which has been widely accepted, as shown in Table 2.4. This system is based on the maturity of carbon black-channel black against acid and neutral carbon black maturity against nature (Antal *et al*., 2003).

Table 2.4: Carbon Black Classification (Donnet and Andries, 1976)

Second Digit	Diameter (nm)	Old Code	Type of Carbon Black
0	1-10	-	-
1	11-19	SAF	Super abrasion furnace
2	20-25	ISAF	Intermediate super abrasion furnace
3	26-30	HAF	High abrasion furnace
		EPC	Easy and medium processing channel
		MPC	
4	31-39	FF	Fine furnace
5	40-48	FEF	Fast extrusion furnace
6	49-60	GPF	General purpose furnace
		HMF	High modulus furnace
7	61-100	SRF	Semi reinforcing furnace
8	101-200	FT	Fine thermal
9	201-500	MT	Medium thermal

2.2.2 Carbon Black usage

The most common usage of carbon black is as a pigment and reinforcing phase in automobile tires. Carbon black also helps conduct heat away from the tread and belt area of the tire, reducing thermal damage and increasing tire life. Carbon black particles are also employed in some radar absorbent materials and in printer toner. In year 2006, the total production of carbon black was about 78932789 kiloton's (Stabik and Dybowska, 2007). About 20% of world production goes into belts, hoses, and other rubber goods. The highest volume of carbon black is used as reinforcing filler in rubber products, especially tires. (Zieliski and Kijelski, 2004).

Carbon black also used to reinforce composite materials, particularly the class of materials known as carbon fiber reinforced plastics. This class of materials is used in aircraft parts, high-performance vehicles, tuner cars, sporting equipment, wind generator blades and gears and other demanding mechanical applications (Singh *et al.*, 2008; Vyas and Singh, 2007). Reinforced carbon-carbon (RCC) consists of carbon fiber-reinforced graphite, and is used structurally in high-temperature applications, such as the nose cone

and leading edges of the space shuttle. Carbon black is also used for the manufacture of dry cells in the electrical industry, the pharmaceutical industry or in the manufacture of paints and varnishes (Singh *et al.*, 2008; Vyas and Singh, 2007).

2.3 Carbon Nano fillers (CNF)

The discovery of carbon nanotechnology begins with discoveries of fullerenes in 1985 and then carbon nanotube in the year 1991. The production cost of the fullerens halted the growth of carbon nanotechnology for a few years. With the advancement in the combustion methods, the production cost is expected to come down by at least one tenth for producing fullerenes (Kotsilkova *et al.,* 2005). While many researchers are trying to look for other alternatives to produce carbon nanoparticles, carbon black is one of the cheapest available sources. Carbon black is being produced all over the world in large quantities and the main drawback of carbon black is its very low crystalline nature (Abdul Khalil *et al.*, 2010; 2012).

The major transformation required is crystallization of carbon black, heating or graphitization process increases the crystallization of amorphous carbon. It known that heating alone does not provide good result for converting carbon black to carbon nano-onions. It is important to get the right quality of nano-onions for a good crystallinity carbon black (Hollertz *et al*., 2011).

On the basis of a number of factors such as low production cost and purity, these carbon blacks can be a starting material for carbon nanotechnology (Chatterjee *et al*., 2012). Researchers have produced carbon nano-onions from carbon blacks upon irradiation of electron beam of high current density of 150A/cm square (Hollertz *et al*., 2011).